

DETERMINATION OF STARCH

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INTRODUCTION

Starch is commonly used in the production of fish jelly products. Its main functions are:-

- (1) as an extender to increase the bulk of production
- (2) as a binding agent

Starch is readily convertible into glucose by hydrolysis either by an enzyme such as diastase or by heating with an acid and its estimation usually depends upon this reason.

The hydrolysed glucose is determined by Somogyi method (D-3). The content of starch in the sample is then back calculated from the content of the glucose.

For the confirmation of presence of starch in fish jelly product, the sample is first heated with water. The starch granules will swell up and burst at about 70°C, resulting in a sticky feel. When iodine solution is added, a characteristic blue colour is developed, due to starch iodide, which is decomposed on heating, but is reformed on cooling.

I SAMPLE PREPARATION

Collect fish jelly product sample (≤ 100 g) and pass 2-3 times through food mincer, or chop very finely and mix thoroughly.

II REAGENTS

- a) 8% potassium hydroxide (KOH) in alcohol.
Dissolve 8 g KOH in 4 ml of distilled water completely and mix with 96 ml absolute alcohol. (Potassium hydroxide must be dissolved in water first as it is insoluble in ethanol).
- b) 50% ethanol.
- c) 2.5% hydrochloric acid (HCl)
14 ml conc. HCl in 186 ml distilled water (Conc. HCl is 35%).
- d) 15% Sodium hydroxide (NaOH)
15 g NaOH (Technical grade) dissolve in 85 ml distilled water.

e) Somogyi solution A

Sodium potassium tartrate (K Na C ₄ H ₄ O ₆ ·4H ₂ O)	90 g	CuSO ₄ ·5H ₂ O	30 g
Trisodium phosphate Na ₃ PO ₄ ·12H ₂ O	225 g	H ₂ O	500 ml
H ₂ O	700 ml		
		KIO ₃	3.5 g
		H ₂ O	100 ml

Make up to 2 litres with distilled water

Keep at room temperature.

f) Somogyi solution B

Potassium oxalate (K ₂ C ₂ O ₄ ·H ₂ O)	90 g
KI	40 g

Make up to 1 litre with distilled water.

Keep at room temperature.

g) 2N H₂SO₄

Conc. H₂SO₄ (60 ml) dilute to 1 litre with distilled water. (Conc. H₂SO₄, 36N, is 95-97 wt %).

h) Starch indicator

Dissolve 1 g soluble starch and 0.1 g salicylic acid in 99 ml distilled water. Boil to dissolve the suspension.

i) Dried KIO₃

Weigh about 2 g KIO₃ and dry in oven at 120°C for 1 hour.

j) 2.5% KI

Dissolve 2.5 g KI in distilled water and make up to 100 ml.

k) 0.05N $\text{Na}_2\text{S}_2\text{O}_3$ solution

Sodium thiosulphate $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ 13g

— Na_2CO_3 0.3 g

Make up to 1 litre with decarbonated distilled water.

Determination of factor, F, of 0.05N $\text{Na}_2\text{S}_2\text{O}_3$

Weigh 1.5 g dried KIO_3 accurately and dissolve in 500 ml distilled water in volumetric flask.

To 10 ml of KIO_3 solution and 10 ml distilled water (BLANK) each add 2.5% KI (20 ml) and 2N H_2SO_4 (20 ml).

Titrate with 0.05N $\text{Na}_2\text{S}_2\text{O}_3$ using starch indicator.

$$\text{Factor, } F = \text{Wt. of } \text{KIO}_3 \times \frac{10}{500} \times \frac{1}{0.0017835} \times \frac{1}{(B - A)}$$

where 0.0017835: conversion factor of 1 ml 0.05N $\text{Na}_2\text{S}_2\text{O}_3$ to KIO_3

A: titration volume of KIO_3 solution (ml)

B: titration volume of blank (ml)

III PROCEDURE

Minced fish ball (10 g)

— Put in centrifuge tube

— 8% KOH-alcohol (50 ml)

Heat at 90-95°C with condenser till starch precipitant occurs (usually 30-40 min.)

Cool down

Centrifuge at 2000 rpm/5 min

— discard supernatant

ppte

— wash with 50% alcohol (25 ml)

Centrifuge

— discard supernatant

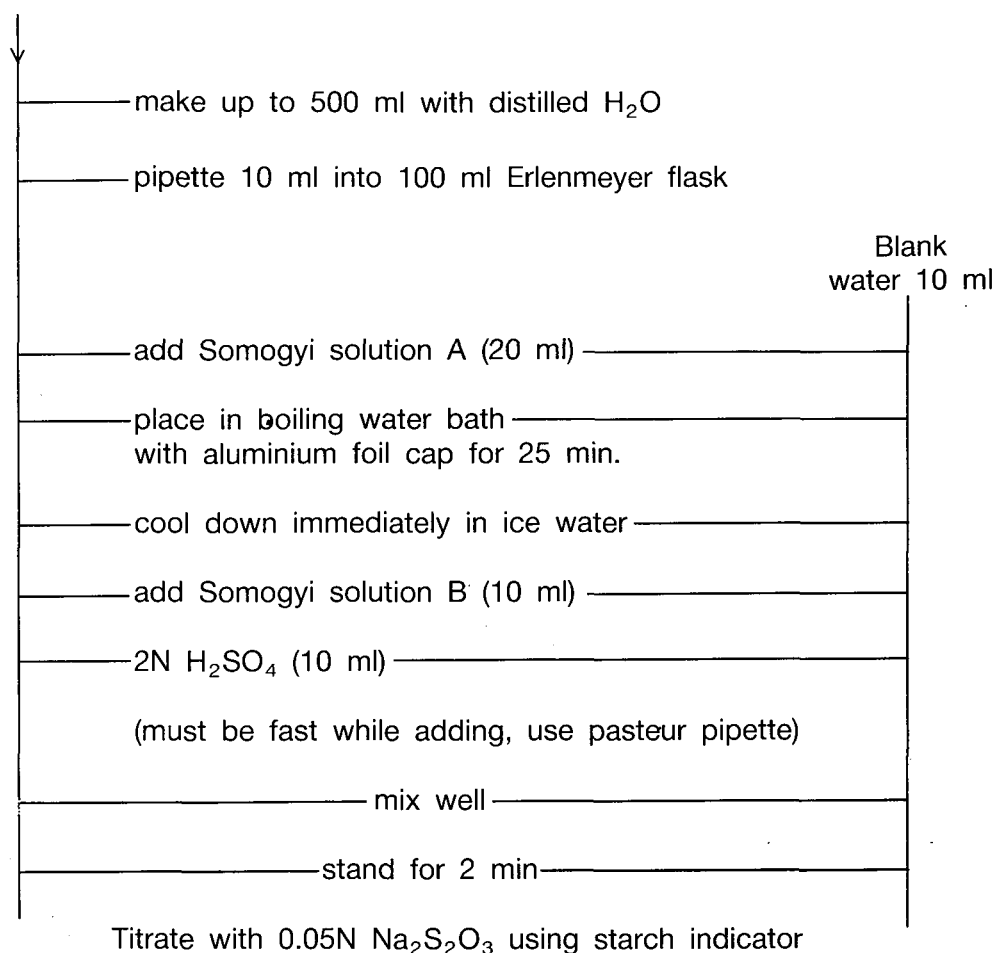
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— transfer ppte into 300 ml Erlenmeyer flask with 200 ml of 2.5% HCl

— boil for 2½ hours in water bath with condenser, cool down

— adjust to pH 6.5-7.0 with 15% NaOH using pH meter

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(Note: colour changes to light blue).

The blank test with distilled water (10 ml) should be carried out simultaneously with the supernatant sample.

IV CALCULATION

$$\text{Starch (\%)} = 0.001499 (B - A) F \times \frac{500}{10} \times 0.9 \times \frac{1}{S} \times 100$$

A = titration volume of sample (ml)

B = titration volume of blank (ml)

F = Factor of 0.05N Na₂S₂O₃

0.001449 = Conversion factor of 0.05N Na₂S₂O₃ (ml) to glucose (g)

0.9 = Conversion factor of glucose to starch.

S = Weight of sample (g).

REFERENCE

Official methods of analysis of the Association of Official Analytical Chemists. 13th Ed. 1980. 24.057:383.